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AN APPARATUS FOR THE DIFFERENTIAL THERMAL ANALYSIS OF EXPLOSIVES

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AN APPARATUS FOR THE DIFFERENTIAL THERMAL ANALYSIS OF ~~EXPLOSIVES~~

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FOREWORD

The work described in this report was funded under SP 271-12.

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ABSTRACT

A differential thermal analysis apparatus has been designed and constructed at the Naval Propellant Plant for use in studying either ground (2.5 g) or right-cylinder (1/2 to 2 in.) propellant samples. Heating rate is 0.50° ± 0.02° C/minute. One feature of this apparatus is the aluminum-foil-covered asbestos oven with a blow-off top. This top plus the fact that the sample holders reduce to dust upon detonation make the replacement of the oven an extremely rare occurrence. Should the oven have to be replaced, however, the cost is only about $100.00.

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FIGURES

AN APPARATUS FOR THE DIFFERENTIAL THERMAL ANALYSIS OF EXPLOSIVES

To study such problems as the reactions and interactions of ingredients, the temperature at which propellant samples will spontaneously (auto-) ignite as a result of heating during processing or storage, and the effect of the mass and geometrical configuration of the sample on the temperature of autoignition, a differential thermal analysis (DTA) apparatus has been constructed at the Naval Propellant Plant to study comparatively large samples of propellant. This apparatus has produced reasonably accurate and reproducible results for the past 5 years; in view of this fact and the queries received, it was felt that it might be of some general interest. For recent reviews of the DTA technique, the reader is referred to Smothers and Chiang, (1) Murphy, (2) and Bohon. (3)

DTA, which has long been used in the ceramics industry, basically comprises the measurement of the absorption or evolution of heat and the determination of the temperature at which thermal pheneomena occur by comparing the behavior of a sample with that of a thermally inert reference substance with which it is being heated. The three components of the typical DTA apparatus are an oven or furnace in which the sample and reference are heated, a device to control the rate of heating, and a means of continuously measuring and recording the temperature of the sample and reference.

Because of the nature of the materials studied, safety and inexpensive replacement were of major consideration in the design. The DTA apparatus described in this report is built around an asbestos oven which can be completely replaced for about $100. The oven is placed in a barricaded room; its large volume, its "easily blown off" top, and the fact that the sample holders reduce to dust upon detonation make the replacement of the oven an extremely rare occurrence.

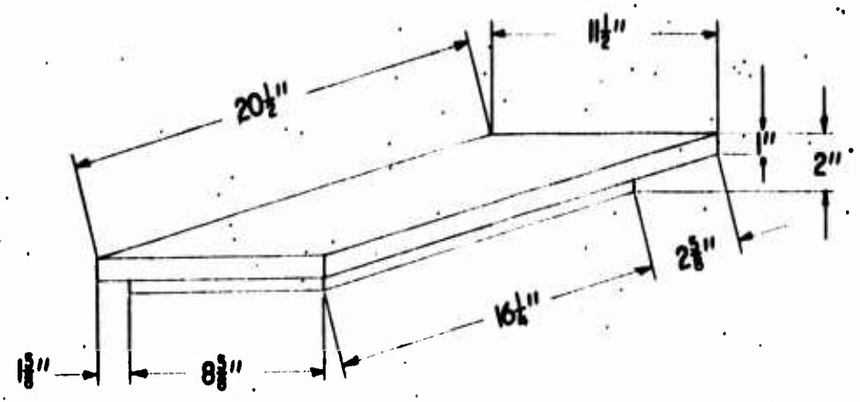
APPARATUS

The heating rate controller employed in this apparatus is the Weston Electrical Instrument Corporation (Tag) Program Controller Model 48352 consisting of a cam-operated programmer which continuously changes the "set point" in one arm of a bridge circuit in the controller. This photoelectric, "on-off" controller senses the oven temperature by means of a copper-constantan thermocouple and makes or breaks the oven heater circuit as required. The end result is a very linear increase in oven temperature over the entire range of the instrument (0° - 350° C).

Since the materials most often studied in this equipment are completely or partially organic in composition, the heating rate adopted as standard is 0.50 ± 0.02° C/minute; this is necessary to minimize the lag between the occurrence and detection of the thermal phenomena which lag is, in large measure, due to the difference between the thermal conductivities of the sample and the calcined Al_2O_3 reference. The slow rate of heating, of course, results in a reduction in the sharpness of the endothermal (minima) and exothermal (maxima) peaks characteristic of the substance being examined. This is partially offset by the use of very large sample sizes, usually 2.5 grams of sample or more. In view of the size of the sample and the nature of the material, the ability to withstand ignition or detonation was purposely designed into the apparatus.

The temperature of the sample (T_S) and of the reference (T_R) are recorded as a function of time on a Brown Electronik six-point recorder; alternatively $\Delta T = T_S - T_R$ is plotted against T_R or T_S on an X-Y Function Plotter.

The oven used in this apparatus was fabricated at NPP from one-half-inch asbestos millboard sheets glued together with sodium silicate; its dimensions are shown in Figure 1. The ends of the oven were nailed to the sides and the outer and inner surfaces were completely covered with a very thin aluminum foil. It has been found better in practice to cover the oven's interior with heavy aluminum foil. The thick end was cut to accommodate the "squirrel cage" (or "snail") blower shown with the other oven components in Figure 2. The heat is supplied by five chromalox heaters connected in parallel; as previously described, the controller-operated switch is placed in one side of the heater power line. The control thermocouple is placed, as shown in Figure 3, at the outlet of the blower



$4\frac{3}{8}$" – DIA. HOLE THRU
$5\frac{1}{8}$" – DIA. COUNTER BORE, $2\frac{1}{4}$" – DEEP (MAX)
$6\frac{1}{8}$" – DIA. COUNTER BORE, 2" – DEEP (MAX)

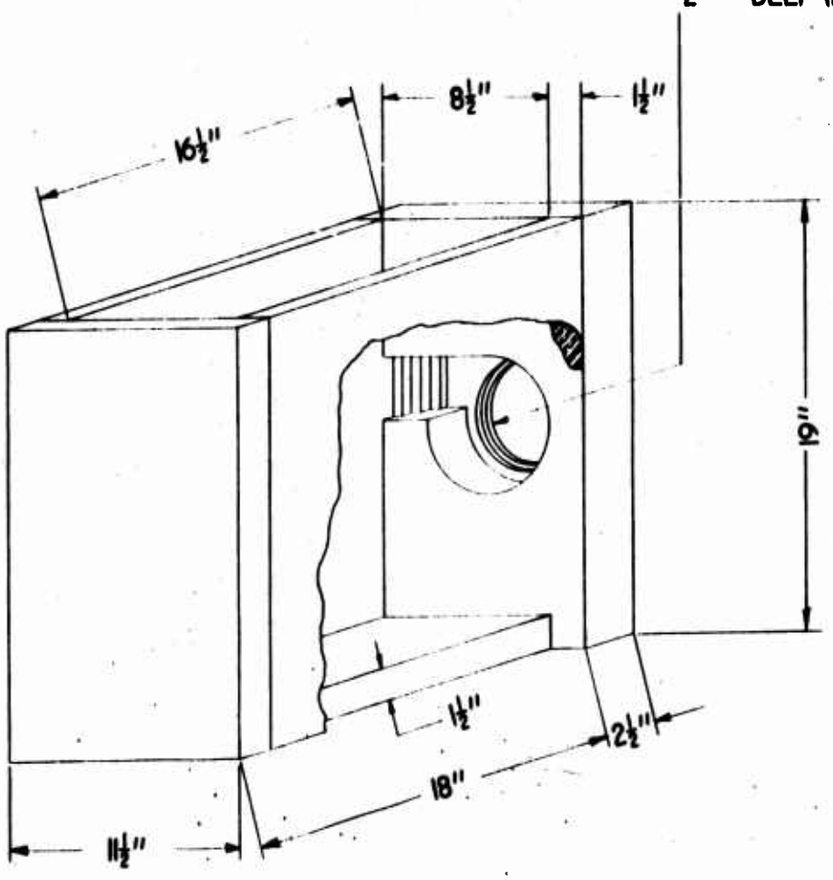


FIGURE 1. ASBESTOS MILLBOARD OVEN AND TOP

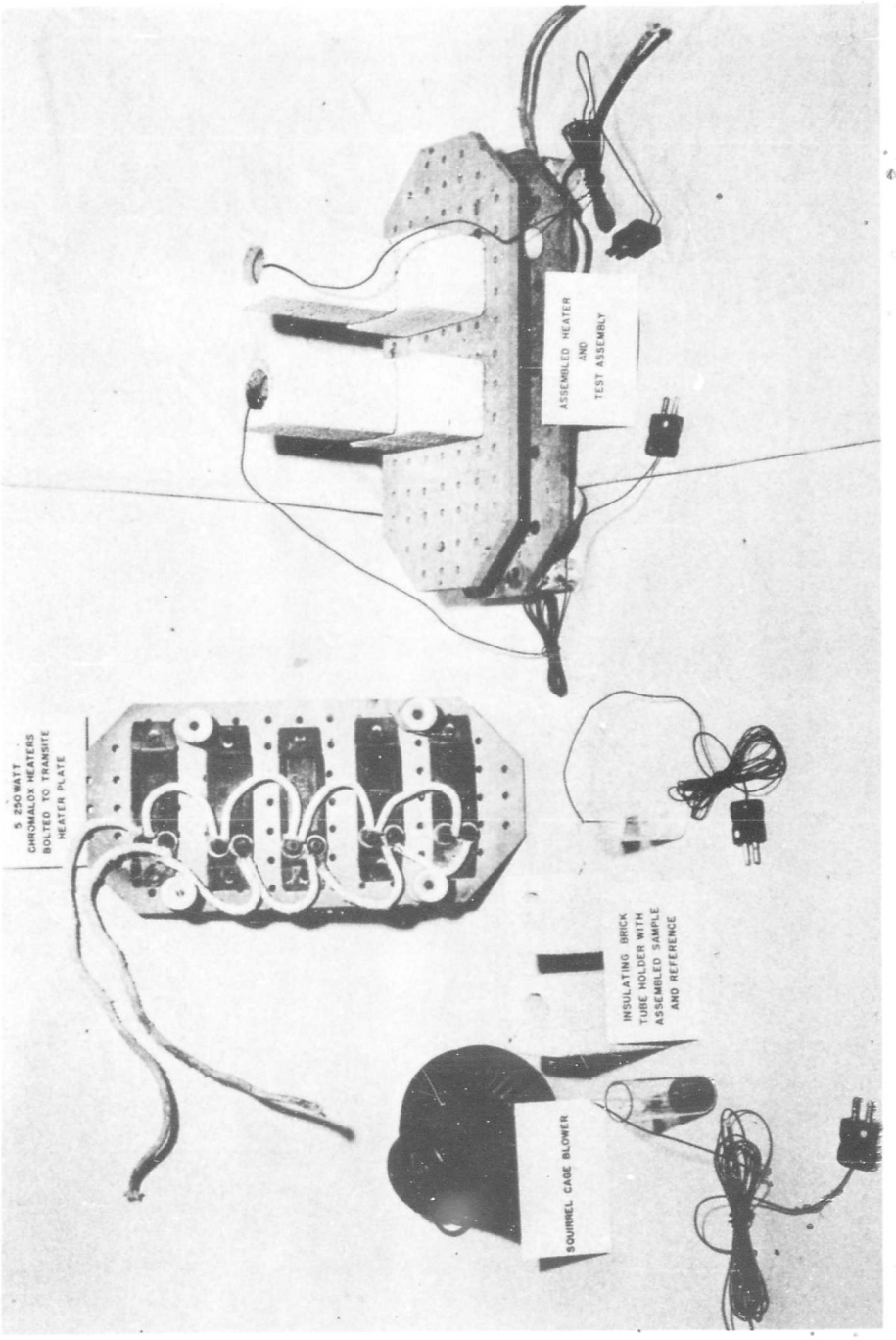


FIGURE 2. COMPONENTS USED FOR HEATING SAMPLES

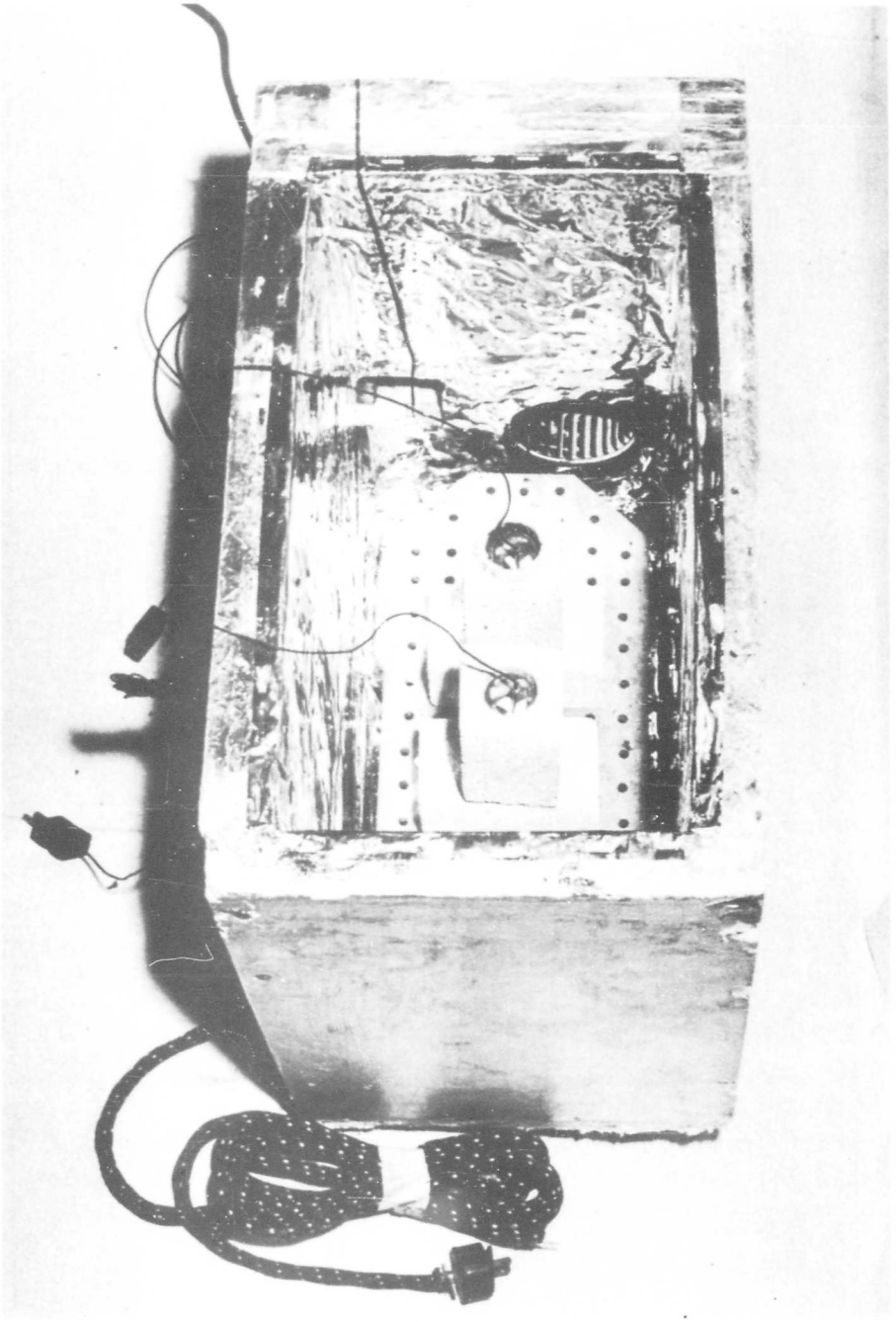


FIGURE 3. DTA OVEN WITH COMPONENTS ASSEMBLED

used to circulate the air rapidly throughout the oven. By this arrangement, heat evolved by a sample is dissipated throughout the entire oven and the control couple is affected immediately causing the heaters to work less frequently. The success of this approach is demonstrated by the fact that no change in rate was observed in a reference being heated with a 2-inch right cylinder of propellant (ca 170 grams) during the exothermal degradation which preceded its ignition.

SAMPLE TREATMENT

The sample preparation and treatment depend on the nature of the sample and the purpose of the investigation. Such materials as inorganic salts are ground with a mortar and pestle to a fine powder and are centrifuged 2 minutes before insertion of the thermocouple. Propellant samples may be standardized as to geometry and packing density by grinding the propellant and sieving it to pass No. 20 and hold on No. 50 U. S. Standard Screens. The material is then dried and a 5-gram sample weighed into a 16-mm Pyrex test tube and centrifuged in a laboratory centrifuge for 2 minutes.

Where grinding would cause separation and stratification of its components, propellant compositions are studied in the form of equidimensional right cylinders measuring from 1/2 to 2 inches. This configuration is also used when the mass effect on T_i is under investigation. A central hole is drilled in the sample cylinder to a depth which is slightly over half its height; this hole is made sufficiently large to just accommodate a 30-gage iron-constantan couple which is sheathed in a glass capillary melting point tube ca 2-mm O.D. Since the thermocouples will interact with inorganic oxidizer salts such as are often found in propellant formulations, the use of the glass sheath has been made a standard practice. Comparisons between the bare versus sheathed couples show that no appreciable lag is introduced in the detection of the thermal phenomena by the use of these thin-walled tubes.

The propellant cylinders are inserted in a glass tube of appropriate diameter (Figures 2 and 4); the thermocouple is positioned by passing the melting point tube through the center of a Teflon gasket which is star-shaped to give the sample free access to the atmosphere in the oven (air). If the sample tends to gas or otherwise react to push out its thermocouple, two small holes can be drilled in

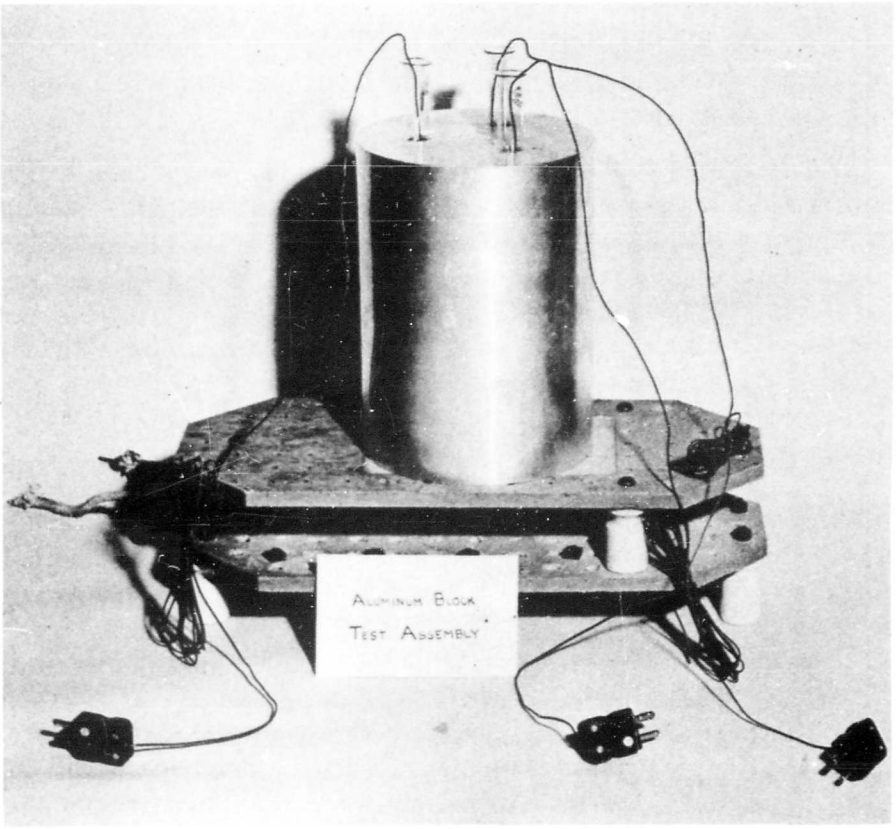


FIGURE 4. ALUMINUM BLOCK TEST ASSEMBLY

the sample tube just at the top of the glass sheath and a fine, insulated iron wire threaded between the insulated couple leads (over top the glass sheath) and through the small holes in the sample tube. The ends of this wire are then twisted together; the restraining action of this wire and of the Teflon gasket keep the thermocouple securely embedded in the sample even during burning.

The calcined Al_2O_3 reference is placed in a similar tube; equal volumes of sample and reference are compared. Chromatographic adsorption alumina (80-200 mesh) was selected as the reference; this was heated at about 650° C for several hours and stored in a desiccator over anhydrous $CaSO_4$ until used.

As shown in Figure 2, the glass sample tube is placed in a ceramic holder (2-1/2 × 2-1/2 × 4-1/2 inches) drilled to accommodate it. This holder is cut from an Armstrong insulating fire

brick (A-16) 9 × 4-1/2 × 2-1/2 inches weighing about 1 pound 14 ounces; this is a very light, porous material which reduces to a powder should the sample detonate.

A variety of aluminum heating blocks have also been used in this asbestos oven to hold the sample tube; one such block shown in Figure 4 is a right cylinder 6 inches in diameter and 8 inches in height, containing three sample wells 4 inches in depth and 5/8 inch in diameter. The centers of these holes are located on the circumference of a circle 3 inches in diameter having the center of the block as its origin; a line drawn from the center of a sample well to the center of the block and thence to the center of an adjacent sample well forms an angle of 120 degrees. This large mass of metal acts as a heat sink and tends to "flatten out" the peaks in the temperature versus time curve due to thermal phenomena occurring in the sample. Large samples will heat or cool the block sufficiently to affect T_R on occasion, and several instances have been encountered in which the heat absorbed by the aluminum block was sufficient to prevent the ignition of a propellant sample which ignited in the ceramic block. In the present design, the aluminum block has the advantage of permitting the simultaneous heating of reference, resin or oxidizer salt, and a propellant containing the resin or oxidizer salt for a simultaneous comparison of propellant versus Al_2O_3, ingredient versus Al_2O_3, and propellant versus ingredient. In general, however, it is felt that the ceramic block produces more realistic results.

There is little to choose between the ceramic and aluminum blocks as far as the thermal symmetry of sample and reference are concerned. The aluminum block is placed in the center of the oven on the top transite plate on which the ceramic blocks (Figures 2 and 3) are also placed. The tube positions in both setups have been shown in Al_2O_3 versus Al_2O_3 runs to be within 0.2° C or less of each other in repeated runs in different ovens over the temperature range 0° - 300° C.

RESULTS

Differential thermal analyses of the organic materials for which this apparatus was designed are available, at present, only in classified publications. The reproducibility of the apparatus can be demonstrated, however, by the DTA curves for NH_4NO_3 and

NH_4ClO_4 shown in Figures 5 and 6. J. T. Baker Reagent grade ammonium nitrate was used without further purification. The ammonium perchlorate was Fisher Certified Reagent grade which had been further purified by recrystallization from aqueous solution to eliminate any possibility of organic contamination.

. . If one takes the point at which ΔT makes its first significant change as the temperature of the phase transition, one obtains temperatures of 35°, 86°, and 126° C, for the three crystalline modifications and 168° C for the melting point of NH_4NO_3. These DTA temperatures agree remarkably well with the accepted temperatures for these phenomena; namely, 32.3°, 84.2°, 125.2°, and 169.6° C. The occurrence of the IV - III phase transition at 35° C instead of 32° C is not unexpected; Hendricks, Posnjak, and Kracek[4] noted that, during heating, this modification is first observed from 36° to 40° C unless both phases are initially present. We observed no significant absorption of heat below 35° C but the phase transition, once started proceeds vigorously. The sample subsequently cools about 0.5° C so that a plot of T_S versus ΔT would show a closed loop in the DTA curve.

The effect of having a large heat sink in contact with the sample is well depicted by the comparison between the firebrick and aluminum block holders. In Figure 5, for example, heat is more rapidly conducted to the sample by the metal so that the endothermal phase transitions are more readily accomplished than in the firebrick. On the other hand, the decomposition of NH_4NO_3 is clearly shown to be exothermal in the firebrick whereas the aluminum block minimizes this effect.

In Figure 6 are shown two typical DTA curves of NH_4ClO_4; the endotherm at 243° C is due to the rhombic-to-cubic crystalline phase transition; the accepted value of which is 240° C. Once again the presence of the aluminum block facilitates the endothermal transformation. It was frequently noted in the DTA examination of commercial grade ammonium perchlorate or even with reagent grade material which had been ground in a Wiley mill ordinarily used to grind organic material that the 240° C endotherm occurred in the middle of a large exotherm. This exotherm is undoubtedly due to an interaction between the perchlorate and the organic compound since its onset is noted at the same temperature for a number of different organic compounds.

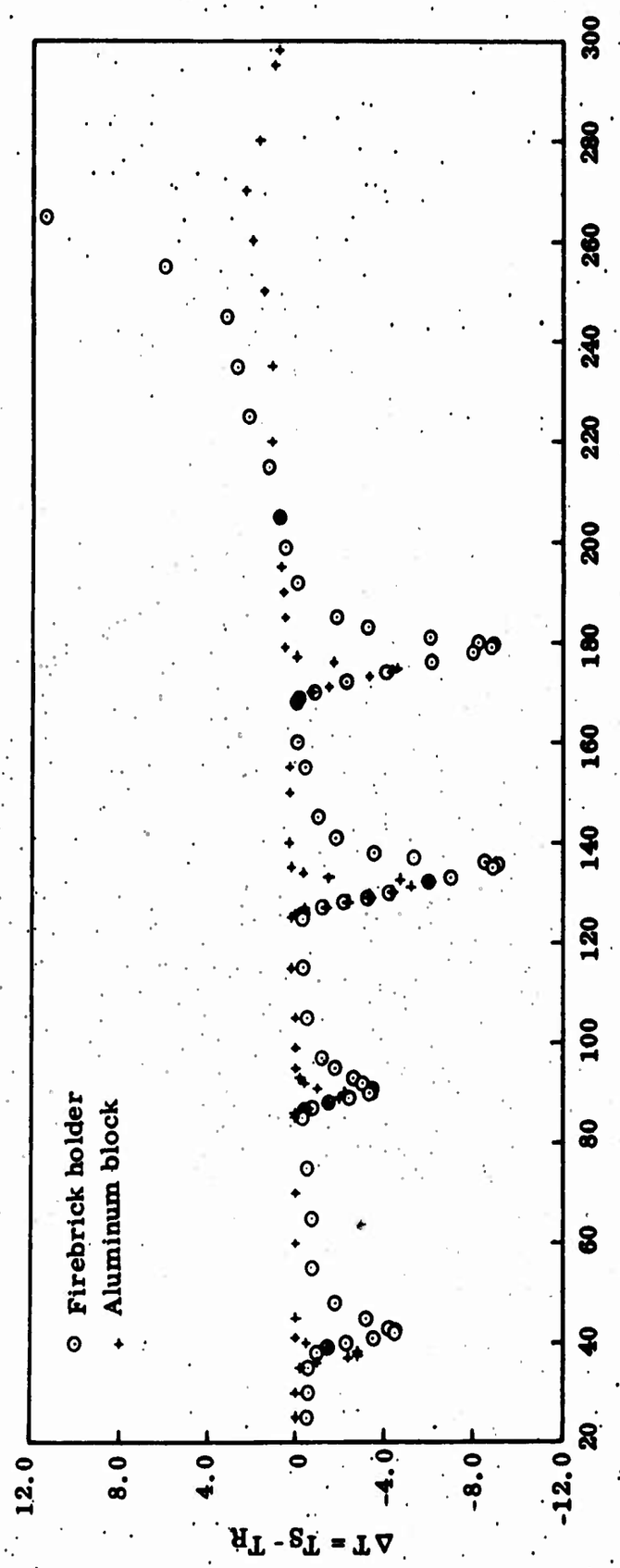


FIGURE 5. DIFFERENTIAL THERMAL ANALYSIS OF NH_4NO_3

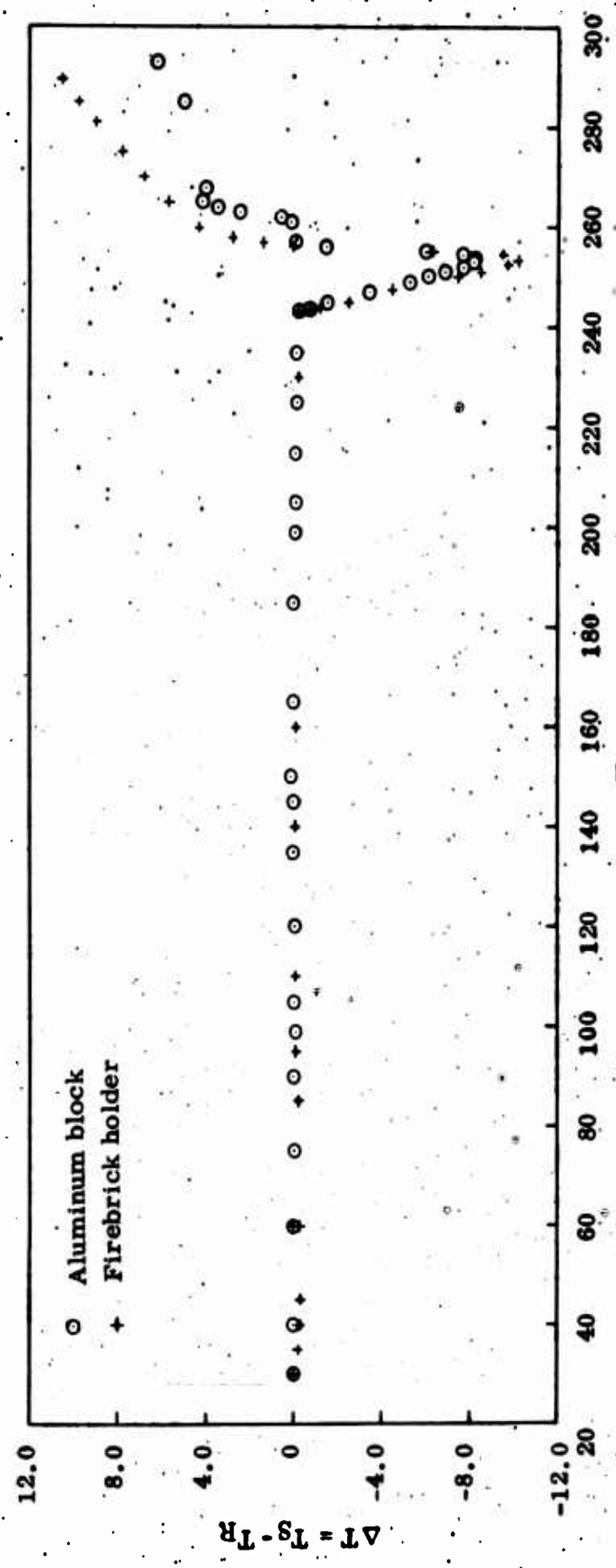


FIGURE 6. DIFFERENTIAL THERMAL ANALYSIS OF NH_4ClO_4

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AN APPARATUS FOR THE DIFFERENTIAL THERMAL ANALYSIS OF EXPLOSIVES (U). By M. E. Baicar, W. G. Gough, and E. F. Hare. 8 December 1961. 13 p. (TR 114)
UNCLASSIFIED

A differential thermal analysis apparatus has been designed and constructed at the Naval Propellant Plant for use in studying either
(over)

1. Differential analysis - Equipment

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ground (2.5 g) or right-cylinder (1/2 to 2 in.) propellant samples. Heating rate is 0.50° ± 0.02° C/minute. One feature of this apparatus is the aluminum-foil-covered asbestos oven with a blow-off top. This top plus the fact that the sample holders reduce to dust upon detonation make the replacement of the oven an extremely rare occurrence. Should the oven have to be replaced, however, the cost is only about $100.00.		ground (2.5 g) or right-cylinder (1/2 to 2 in.) propellant samples. Heating rate is 0.50° ± 0.02° C/minute. One feature of this apparatus is the aluminum-foil-covered asbestos oven with a blow-off top. This top plus the fact that the sample holders reduce to dust upon detonation make the replacement of the oven an extremely rare occurrence. Should the oven have to be replaced, however, the cost is only about $100.00.	
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